



LABNOTES

Fall 1999



The newsletter of the Wisconsin Laboratory Certification and Registration Program
Program Information: (608) 267-7633 Telefax: (608) 266-5226

WDNR to Apply for NELAP Recognition

Diane Drinkman, Laboratory Certification Program

This August, George E. Meyer, Wisconsin DNR Secretary, authorized the Laboratory Certification Program to begin the process of preparation for applying to the National Environmental Laboratory Accreditation Program (NELAP) to become a recognized accrediting authority. Once the Department has become accredited it will be able to accredit laboratories according to the standards set by the National Environmental Laboratory Accreditation Conference (NELAC).

“We plan to submit an application to NELAP by May 2000, and if it gets approved and all goes well, we will start performing NELAC on-site assessments of laboratories by the spring of 2001”, says Alfredo Sotomayor, Senior Audit Chemist with the Laboratory Certification and Registration Program.

TWO TIERS: ACCREDITATION AND CERTIFICATION

The Department intends to establish a two-tiered system for regulating laboratories performing compliance environmental work based on recommendations from the NELAC Advisory Committee. Municipal and industrial laboratories that only perform Clean Water Act work (wastewater analysis) will be covered under a modified Chapter NR 149, of the Wisconsin Administrative Code that eliminates the registration option and treats them as certified.

All commercial laboratories and those non-commercial laboratories doing work in support of the Safe Drinking Water Act (SDWA) or the Resource Conservation and Recovery Act (RCRA) will be required to participate in an accreditation program based on the NELAC standards. Accreditation will also be offered for completing CWA work for these laboratories so that they do not have to comply with two sets of standards. Although all laboratories must be either certified under ch. NR 149 or accredited by the NELAC standards, the ch. NR 149 laboratories have the option of joining the NELAC group voluntarily.

(see WI to Apply to NELAP, page 2)

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LabNotes - Newsletter of the Laboratory Certification Program

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This newsletter is intended to present current information and issues to certified and registered laboratories. This newsletter does not establish policy for the Department.

(WI to Apply to NELAP, continued from page 1)

“The Department has made great efforts to involve the regulated community as we considered applying to NELAP and as we envisioned what type of a system would be best for us and our laboratories”, says Sotomayor. “After forming a Technical Advisory Committee, convening focus groups, and meeting with interested organizations and selected laboratories, we understood that a two-tiered system was the most popular alternative. We have also heard loud and clear that laboratories will need help with the transition to a NELAC program.”

HELP TO LABORATORIES

“We are committed to providing several aids to laboratories to manage upcoming changes. We plan to craft primers of the Standards, model QA Plans and SOPs, and are exploring establishing ‘model’ NELAC labs and pre-accreditation audits”, Sotomayor states. “Applying to NELAP would help strengthen and refine the Department’s documentation systems and accountability to regulated laboratories. There is much work that needs to be done in a relatively short period of time, but we are all very excited about this great opportunity.”

For more information about the Wisconsin Laboratory Certification Program’s NELAC implementation plans, contact Alfredo Sotomayor at (608) 266-9257 or at sotoma@dnr.state.wi.us.

ADMINISTRATIVE INFORMATION

Lab Certification Web Site Trends

Ron Arneson, Bureau of Integrated Science Services

The WDNR Web Master runs a report once a month to evaluate usage of Department web sites. Reports are issued by Bureau; the Laboratory Certification and Registration Program is included in the Bureau of Integrated Science Services (ISS). For the period 7/31/1999

to 8/31/1999:

- The Lab Cert Homepage is the most viewed;
- Most users go directly to the Lab Cert home page (without using other pages to get there);
- The most accessed directory for ISS is the Lab Cert directory;
- The most downloaded files are Lab Cert files;
- The 2nd most viewed page is the ISS Home page;

RECENT ADDITIONS TO THE

LABORATORY CERTIFICATION WEBSITE

- QA Manual for a Small Wastewater Lab
- Training Handouts from the Spring of 1999 QA/QC Course
- Updated application for Large Laboratories
- New Application for In-State WWTP Labs
- Laboratory Toolbox
- Web Site Map

Certification Program Status Report

In August the Certification Standards Review Council was briefed on the numbers of audits, reports issued, closed cases and enforcement actions performed by the Laboratory Certification and Registration Program in recent years.

For **Registered** laboratories (approximately 360 of the 510 laboratories in the program):

Fiscal Year	Audits	Reports	Closed Cases	Enforcement Actions
1996	62	55	48	0
1997	74	65	61	1
1998	114	115	105	6
1999	113	105	89	10
2000 Projection	140	120-140	120-140	5-10

For the **Certified** laboratory community (approximately 150 laboratories):

Fiscal Year	Audits	Reports	Closed Cases	Enforcement Actions	Unseen Labs
1998	44	39	58	3	6
1999	40	42	47	6	5
2000 Projection	25	28	30	5	0

In addition, the standards Review Council was given the priorities for selecting certified laboratories to audit during fiscal year 2000:

- Laboratories that have never been audited;
- Applications that trigger audits within 90 days;
- Laboratories with oldest priorities for revisits;
- Laboratories with problems known to the Department to certification staff; and
- Laboratories with chronic reference sample failure.

Staff Changes

Brenda Howald was recently hired as a ½ FTE (Full-time Employee) Regional Certification Officer for the South Central Region. She had served in a similar capacity as a Limited Term Employee for the past two years.

Brenda's experience in environmental laboratory analysis, Quality Assurance and wastewater permitting is a perfect match for the position. The next time you see Brenda, please join us in congratulating her on her new position!

Also, Gül Uludöğün recently accepted a position with USDA in Illinois. Gül's efforts with reference samples will be missed.

Nominations for 2000 Lab of the Year Awards Sought

Greg Pils, Laboratory Certification Program

The laboratory certification program is seeking nominations for the 2000 Registered Lab of the Year Awards. The awards are presented annually to recipients in two categories: Small Facility (wastewater treatment plant labs with flows less than 1 mgd, or labs that perform a limited array of tests), and Large Facility (wastewater treatment labs with flows greater than 1 mgd, or labs that perform a wider array of more complex tests).

Nominees for Lab of the Year must be *registered* facilities located in the State of Wisconsin. Certified laboratories will not be considered. Anyone, including DNR staff, can nominate a laboratory for one of the awards, but laboratories may not nominate themselves. There is no limit on the number of times that a laboratory may be nominated, and a laboratory may be nominated (or presented) an award in consecutive years. Awards will be presented to the winners at the March 2000 meeting of the Natural Resources Board.

To nominate a laboratory for 2000 Lab of the Year, contact Greg Pils at (608) 267-9564 or pilsg@dnr.state.wi.us for a nomination form. Completed nomination forms must be received by December 31, 1999.

WSLH Proficiency Testing Program

Barb Burmeister, WSLH Proficiency Testing Program

The Wisconsin State Laboratory of Hygiene has been a provider of environmental proficiency testing samples since 1986, helping laboratories meet state certification requirements.

As you know, the EPA discontinued the WP, WS and Discharge Monitoring Report Quality Assurance (DMRQA, for WWTPs with >1 MGD flow) laboratory performance evaluation (PE) programs in 1998. The discontinuation of the EPA programs has had minimal effect on the operation of the Wisconsin DNR Laboratory Certification and Registration Program because the Wisconsin DNR already accepts PE results from several government and private providers in lieu of the EPA. The Wisconsin State Laboratory of Hygiene continues to be an approved provider of PE samples for the Wisconsin DNR Certification and Registration Program.

The WSLH Proficiency Testing Program was not among the first group to apply to the National Institute of Standards and Technology (NIST) for accreditation of PT providers. This means that laboratories cannot use WSLH samples to meet the requirements of the DMRQA program.

If you have questions about the WSLH Proficiency Testing Program, contact Barb Burmeister at (800) 462-5261, ext. 107 or burmie@mail.slh.wisc.edu.

Detection Limits — Two Suggestions When MDLs Are Unrealistically Low

Matthew Roach, WI State Laboratory of Hygiene

The calculation of detection limits is currently the subject of scientific as well as regulatory debate. Most laboratories use the MDL procedure developed by the US EPA and described in 40 CFR Part 136, Appendix B (also see WDNR document "Guidelines for Calculating Detection Limits"). This procedure requires determining the standard deviation of seven or more replicates at a concentration near the MDL estimate. Although some researchers believe the procedure is statistically flawed, it is still widely used and often required. Further, there is no widely accepted alternative.

As many of you know all too well, using EPA's MDL procedure sometimes leads to detection limits which are impossibly low, particularly for very precise analyses. NR 149 allows analysts to use professional judgment to determine whether a substance has been detected. This judgment can be used when your experience tells you that the calculated MDLs are just not possible, provided that a reasonable justification is documented. While the type of justification is not specified in NR 149, two approaches for determining "common sense" detection limits (and their justifications) are given below.

Using Blank Information to Determine an LOD The first approach uses accumulated "method blank" data to establish a detection limit. This technique has been used for many years by the U.S. Food and Drug Administration (FDA). Using a good population of (20 or more) method blank data, the analyst calculates their mean and their standard deviation. A reasonable estimate of the detection limit can then be calculated by adding three standard deviations to the mean. Once you have obtained this estimate, you can check how reasonable it is by calculating a limit of quantitation (LOQ) multiplying the detection limit estimate by three. If analytical standards at concentrations greater than (but close to) this LOQ can be determined with reasonable

accuracy, you have "validated" your "common sense" LOD.

Back Calculation from a Good LOQ A second approach involves determining the LOQ and then back calculating the detection limit. To estimate the LOQ, serial dilutions are run at ever lower concentrations until you arrive at the lowest concentration that still yields reasonable accuracy. With the LOQ established, the detection limit is estimated by dividing the LOQ by three (based on generally accepted detection limit conventions). A standard analyzed just above the LOD estimate should be detectable.

Other Considerations Unfortunately, both of the approaches above imply that standards run at or above the LOQ can be determined with reasonable accuracy. What constitutes reasonable accuracy is obviously subjective. A confidence measure for some metals analysis may be the true value +/- 15%, while for volatiles it may be closer to 30%. Analysts may use the regulatory method and scientific literature, as well as their own experience, as guidelines.

You should clearly document and understand whatever alternative you choose. If you do, then chances are an auditor will be convinced that you have proceeded in a reasonable fashion. This documentation is crucial for compounds that have very low or unachievable regulatory limits, because for these substances, the detection limit de facto becomes the regulatory limit. Ideally, your new detection limit will be *above* the impossibly low MDL but *below* any regulatory limit for the analyte. If not, then perhaps a different analytical technique is in order.

Thanks for QA Training Assistance

In March and April 1999, the Laboratory Certification and Registration Program presented successful training sessions in Quality Assurance for wastewater laboratories. Outreach is something we've talked about for years, but 1999 finally saw all our plans come to fruition. These sessions would not have been possible without

the assistance of *George Bowman* (WI State Laboratory of Hygiene), and *Kay Marshall* (Wisconsin Rural Water Association).

George provided invaluable assistance with technical knowledge, developing the training and as an instructor. These sessions would not have possible without Kay Marshall and the Wisconsin Rural Water Association. The WRWA staff coordinated dates, training facilities, distributed flyers to their members and handled registration. Kay provided invaluable support and assistance at each of the training seminars. Perhaps more importantly, she has tirelessly bridged the gap between the training and performance in the laboratory since that time.

The Department's mission calls for "partnering" with organizations such as the State Laboratory of Hygiene and the Wisconsin Rural Water Association. The success of this training program has reinforced the value of such partnerships.

GEMS: What the WDNR Does With Groundwater Data

Have you ever wondered how the Wisconsin Department of Natural Resources (WDNR) manages electronic groundwater monitoring data submittals? The Bureau of Waste Management has a central database, the Groundwater and Environmental Monitoring System (GEMS), that currently stores over 6 million sample results collected from 526 landfills. The transition to electronic data submittal in July of 1996 has greatly improved our ability to enter data directly into GEMS, eliminating transcription errors during data entry, and has allowed us to capture more information about a sample (i.e. quality control flags, analysis method information, and lab certification identification numbers). Today almost all facilities (excluding the very small sites for which the transition wasn't cost-effective) are successfully submitting data electronically.

There are over twenty reports available in GEMS

that allow staff to analyze the groundwater data. These reports provide staff with the ability to summarize water quality data, evaluate trends, highlight values that exceed groundwater standards, quickly assess site conditions, summarize monitoring requirements and well construction information. Almost all of the reports utilize the qualifiers, quality control flags, LODs, LOQs, and reporting limits that are provided. A report dedicated to displaying QA/QC data provides a printout of quality control flags, the laboratory's certification number, and method codes associated with the sample results aids in data analysis.

As we look to the future, we hope to improve the efficiency, ease, and accuracy of data submittal and screening, and information that is captured. We also are exploring how the submittal procedure can be simplified – suggestions have included e-mail, using ASCII delimited text files, or creating a routine to submit data via the web. We look forward to working with the laboratory community to develop and implement electronic data acquisition practices that will work for all of us.

WASTEWATER INFORMATION

Municipal Wastewater Laboratory Forum

Rick Mealy, Laboratory Certification Program

QUALITY ASSURANCE PLANS

If you were to be audited today, could you produce your laboratory's Quality Assurance (QA) Plan? *Before* the audit is over? If you have a QA Plan for your laboratory, raise your hand (*yeah, right now...I know it'll look a bit strange if someone walks in, but work with me here*)... if your QA Plan is the little red booklet obtained from the DNR 10 years ago, put your hand down. Is your QA Plan the "blue" book titled "*Quality Assurance Document for a Small Wastewater Laboratory*" from 1992 or the "green" 1999 version? If so, put your hand down

as well.

We've lost quite a few hands. Now, for the *piece de la resistance*, if you refer to your QA Plan daily, follow it routinely to perform and evaluate analyses, and have religiously updated it to reflect the changing procedures and Code requirements, reach back with your hand and pat yourself on the back. If not, lower your hand. All you folks who aren't busy patting yourselves on the back have some work to do. Based on what we're finding in on-site evaluations, too many labs aren't using their QA Plan the way they are intended.

When the Laboratory Certification program started in 1987, having a QA Plan was a strongly encouraged. What I've found however, is that not many people realize that effective December 1, 1992, it became a *Code requirement to have and use a QA plan*. You *will* be asked for yours during an on-site evaluation. If you don't have one, expect to see it cited in your audit report. Worse, if not having a QA Plan was mentioned in a previous audit report, and you *still do not have one*, you might be facing enforcement action.

Your QA Plan does not need to be a Pulitzer prize-winning novel, but it *does* have to reflect method and Code requirements. Above all, it must detail how the laboratory evaluates its performance and identifies the circumstances when corrective action must be taken. In preparing your QA Plan, you must ensure that it meets the requirements for recordkeeping and quality assurance in NR 149.

The section devoted to sample handling (including sample points, preservation, receipt and storage) might only be one page long. For each analysis the laboratory performs, summarize the essential elements, required QC (i.e., calibration, replicates, control limits), and what action must be taken if you fail the QC requirements.

If you need assistance in developing your QA Plan, don't hesitate to contact your Regional certification auditor or myself at (608) 264-6006 or via email mealыр@dnr.state.wi.us.

Seeding BODs

Brenda Howald, South Central Region Auditor

For what seems to be quite a simple procedure, seeding of samples for BOD testing can be subject to many complicating factors. The type and number of microorganism in your seed can greatly affect the BOD results of seeded samples. Whether you purchase your seed commercially or obtain it from a wastewater treatment plant there are pitfalls to be avoided. Once you know the ins and outs of seeding BOD samples you will find that with a little care it can be a reliable straightforward procedure.

Raw Domestic Wastewater as Seed. For wastewater treatment plants settled raw domestic wastewater is a good source of seed. It contains bacteria that oxidize carbonaceous organic matter, has few if any nitrifiers and is rich in ammonia and organic nitrogen. However, care must be taken in obtaining the grab for the seed source. It must be collected upstream of any return from secondary treatment processes such as activated sludge, trickling filters, or rotating biological contactors. Otherwise, the collected "raw" is likely to have a large population of nitrifiers due to the nitrification that occurs in secondary treatment processes. The outcome is a sample or standard result that can be biased high due to the oxidation of nitrogen present. It is also important to take the grab at a time well before testing samples for BOD in order to allow the solids to settle. A raw domestic wastewater contains soluble and particulate organics. As every wastewater treatment operator knows, suspended solids in a plant influent are highly variable in size and quantity. Settling the influent before use minimizes the variability in the seed.

Commercially-Available Seed. There are commercial seeds available, which are subject to a different set of problems. You need to find a commercial seed that contains heterotrophic bacteria, which will only oxidize carbonaceous organic matter. Then the instructions for seed preparation need to be carefully followed to provide you with a good seed source. Most of the instructions suggest using dilution water to re-

hydrate the seed. They are referring to aerated distilled water containing the nutrients used in the BOD test. Preparation of the seed with distilled water alone can result in a low concentration seed or one that performs poorly. The exposure of the microorganisms to distilled water can shock the bugs and cause cell damage due to the lack of dissolved solids. If the water is not aerated there can be insufficient dissolved oxygen available to the microorganisms. The trick to successfully re-hydrate your bugs includes time, nutrients and oxygen to provide you with a good source of microorganisms.

Now that you have a good seed source, you need to make sure that you will have enough bugs in your seeded samples and standard. *Standard Methods* includes a range of 0.6-1.0 mg/L depletion in the seeded sample due to the seed (your seed correction factor). A low bias is usually due to not enough seed. There aren't enough bugs to get the job done in 5 days. Too much seed reduces the dissolved oxygen available to your diluted samples—you are more likely to end up with residual DO readings less than 1 mg/L.

Another critical point when using seed is determining the correction factor to use in your sample result calculations. First, you need to set up a seed control blank using a series of dilutions to determine the BOD of the seed. Then use all the bottles with acceptable depletions to calculate the DO depletion per milliliter of seed. (The acceptable depletion criteria require a DO depletion of at least 2 mg/L, and a residual DO of at least 1 mg/L.) This average value can be multiplied times the volume of seed added to the sample or standards to determine the seed correction factor.

Successful seeding comes down to a few simple factors- use the right kind and amount and keep those bugs happy, be careful with calculations and determining your correction factor. Put it all together and you will get excellent results.

Electronic WPDES Data Form Available

Tom Mugan, Bureau of Watershed Management

Wastewater permit applicants may be asking contract laboratories for electronic reports that follow the WDNR Data Reporting Form. Permit application instructions indicate that laboratory reports may simply be attached if they follow the same format as the Data Reporting Form.

We encourage this approach as it eliminates permittees having to reformat data onto the application form (and eliminating transcription errors). In addition, this brings us one step closer to electronic wastewater data reporting.

Data Reporting Forms are available from Tom Mugan at (608) 266-7420 or via email: mugant@dnr.state.wi.us. Donalea Dinsmore of the Laboratory Certification Program can also supply the file. Contact her at: (608) 266-8948 or dinsmd@dnr.state.wi.us.

BOD Training Offered

After the tremendous success and participation at QA/QC training last spring, Rick Mealy and George Bowman have developed a day-long training session for laboratories conducting BOD analysis.

COURSE OBJECTIVES

- The importance and use of BOD tests
- Common problems and remedies
- Calibration, seeding, and maintenance
- Method details and QA/QC
- Troubleshooting
- Record keeping
- Tools to successfully undergo audits

Unfortunately, the timing of the publication of LabNotes was delayed so that the information was not available to the laboratory community.

Course materials will soon be available on the Lab Cert website or by contacting the program at (608) 267-7633 or LabCeD@mail01.dnr.state.wi.us.

REGULATORY UPDATE

FEDERAL REGISTER

REVISIONS TO THE UNREGULATED CONTAMINANT MONITORING REGULATION FOR PUBLIC WATER SYSTEMS; FINAL RULE (9/17/99) EPA has set an effective date of January 1, 2001 for the Unregulated Contaminant Monitoring Regulation. In this final rule, chemical and microbiological contaminants were categorized in three lists, based on present analytical method availability, contaminant stability in sampling protocols and methods under development. Chemical contaminants included in List 1 include: 2,4-dinitrotoluene, 2,6-dinitrotoluene, 4,4'-DDE, Acetochlor, DCPA di acid degradate, DCPA mono acid degradate, EPTC, Molinate, MTBE, Nitrobenzene, Perchlorate, and Terbacil.

Refer to the *Federal Register* for List 2 and 3 chemical constituents and microbiological contaminants and applicability of the monitoring requirements. Additional information can be obtained from the SDWA Hotline (800) 426-4791 or EPA's SDWA webpage: www.epa.gov/safewater/.

WI ADMINISTRATIVE CODE

CHAPTER NR 140 GROUNDWATER QUALITY

Boron: Last December, Chapter NR 140, Wisconsin Administrative Code was revised to establish a new health-based groundwater standard for boron. The new standard becomes effective January 1, 2000.

The new standard for boron, as an indicator parameter in NR 140.20. Table 3, will be:

Preventive Action Limit (PAL): 190 µg/L
Enforcement Standard (ES): 960 µg/L

Facilities monitoring for boron will need to insure that the analytical method their lab uses has a limit of detection and limit of quantitation below the new preventive action limit. To prevent

contamination, avoid using glass when collecting samples, use polyethylene sample containers and PTFE vessels for digestion.

The colorimetric method is not recommended due to the likelihood of contamination. ICP methods tend to be more reproducible and detection limits are 5 - 10 µg/L.

Questions about the new boron standard can be addressed to Jack Connelly at (608) 267-7574 or via email at connej@dnr.state.wi.us.

Toluene and Xylenes: In August, public hearings were held by the Department to address revisions of the toluene and xylenes standards in ch. NR 140, Groundwater Quality. The proposal will revise the preventive action limit (PAL) to address taste and odor concerns associated with these substances and enforcement Standard (ES) to comply with federal drinking water regulations.

The proposed revisions for **toluene:**

Preventive Action Limit (PAL): 0.2 mg/L
Enforcement Standard (ES): 1 mg/L

and for **xylenes:**

Preventive Action Limit (PAL): 1 mg/L
Enforcement Standard (ES): 10 mg/L.

Adoption was requested at the October Natural Resources Board meeting, and are expected to become effective early next year.

Questions about the proposed toluene and xylenes standards can be addressed to Steve Karklins in the Bureau of Drinking Water and Groundwater at (608) 266-5240 or via email at karkls@dnr.state.wi.us.

Ignitability of Solids – Update

Dave Parsons, Bureau of Waste Management; Diane Drinkman, Bureau of Integrated Science Services; Alfredo Sotomayor, Bureau of Integrated Science Services

“...hit it with a hammer, throw a match on it, spray it with water, and see what happens.” If you have a very good memory (or good archives), you may remember that an old issue of LabNotes suggested those high-tech procedures, very tongue in cheek, for determining whether a solid that contained no liquid meets the ignitability characteristic (D001). The truth is that paradoxically, although an ignitable solid is legally defined, there are no required test methods for determining if the criterion is met. Method 1030, ‘Ignitability of Solids’, finalized in Update III of SW-846, contains this disclaimer, underlined: *“...this method may be used, but is not required, to determine whether a solid waste ‘when ignited, burns so vigorously and persistently that it creates a hazard’.”*

Solid Ignitability Characteristic Definition NR 605.08(2)(a)2 defines a solid as exhibiting the characteristic of ignitability if: “It is not a liquid and is capable, at a temperature of 25° C and a pressure of one atmosphere, of causing fire through friction, absorption of moisture or spontaneous chemical changes **and**, when ignited, burns so vigorously and persistently that it creates a hazard.”

Is the Waste Solid? To establish whether a solid is ignitable one must first determine whether the material in question is a solid. One usually determines this indirectly, by ascertaining that no liquid can be expressed from the material. The definitive procedure for determining this is the TCLP (Method 1311) filtration. If one obtains a liquid by the simpler Paint Filter Liquids Test (Method 9095), then that liquid can be used to evaluate ignitability and corrosivity. Wastes that yield no liquid by the Paint Filter test should be submitted to the TCLP filtration to determine if they indeed contain no liquid and thus are only solid.

Once a waste is determined to be only solid, then

it must be evaluated against the two parts of the ignitability definition joined by the conjunction ‘AND’. **Both parts of the definition must be met before you determine that a solid is an ignitable hazardous waste.** Let’s deal with the first part.

Can the Waste Cause Fire Through Friction, Absorption of Moisture, or Spontaneous Chemical Changes? A match is an example of a material that causes fire through friction. On the other hand exposing a solid to a lighted match does not prove that the solid would cause fire through friction. As we said, no specific methods are required to determine these conditions. However, DOT hazardous material transportation rules (contained in 49 CFR Part 173.124) are helpful here, at least for the last two conditions.

“Causing fire through...absorption of moisture” can be evaluated by noting whether the solid becomes spontaneously flammable or gives off flammable gas at a rate greater than 1 L/kg of solid per hour when tested according to the ‘UN Manual of Tests and Criteria’.

“Causing fire through...spontaneous chemical change” can be evaluated by noting whether the solid: 1) is pyrophoric and ignites even in small quantities without an external ignition source within five minutes of coming into contact with air or 2) is a self-heating solid (without an energy supply) that when in contact with air ignites spontaneously or reaches 200° C during the 24 hour test period when tested according to the ‘UN Manual of Tests and Criteria’.

Does the Solid, When Ignited, Burn So Vigorously and Persistently...? This is the second part of the definition. Method 1030 is a valid alternative to the facetious ‘throwing a match on it’ when the solid in question is a paste, a powder, is granular, or can be cut into strips. The method helps to determine whether these types of solids could burn vigorously and persistently. You do not have to use this procedure to evaluate the second part of the ignitability definition, but if you do, you would be using a standard, defensible, and reproducible

protocol.

For Solids, It Takes Two Remember that solids have the hazardous waste ignitability characteristic only when they meet **both** parts of the ignitability characteristic as outlined above. This simplifies the determination for solids that do **not** burn vigorously and persistently when tested according to Method 1030. Since both parts of the definition must be met, a solid that does not burn vigorously and persistently could not be an ignitable hazardous waste.

So for the hazardous waste ignitability characteristic, it may be easier to conclude that a solid is NOT ignitable than to prove that it is.

For more information, contact Dave Parsons at (608) 266-0272 or parsod@dnr.state.wi.us.

The authors thank Dan Elwood of Commonwealth Technology, Inc., Baraboo, WI for suggesting this article.

NELAC V HIGHLIGHTS

The annual NELAC Conference took place in June 1999, in Saratoga Springs, NY. The main event was the recognition of eleven accrediting authorities by NELAP: CA, CO, FL, IL, KS, LA, PA, NH, NJ, NY, UT. NELAP estimated that these accrediting authorities would have enough resources to audit their own in-state laboratories and any other laboratories located in states that have not applied for recognition. Laboratories must apply by November 2, 1999 to be guaranteed a NELAC assessment in the next two years and be potentially included in the first group of accredited laboratories. Other developments:

- The 1999 Standards will be used to accredit the first set of laboratories.
- The first set of accredited laboratories, whether in the interim or in full, will be announced on July 1, 2000. Interim status will be “hidden” until July 2001, to avoid the

potential competitive advantage of the laboratories successfully assessed on-site.

- Mobile laboratories, all requiring separate accreditation, are now covered under NELAC. A definition of a mobile laboratory that can be covered by a parent’s accreditation is in the works.
- The curricula for assessor training courses are soon to be completed by a contractor.
- Training in ethical practices is now required of all laboratory personnel.
- The Accrediting Authority Review Board has been instituted, but its scope, composition, and duties are under review.

The NELAC interim meeting will be held in Washington DC, in December 1999. For up-to-date standards, meeting summaries and proposed revisions, visit the NELAC website (<http://www.epa.gov/ttnnela1/>).

THE AUDITOR’S CORNER

Raw Like Sushi ¹

(PART I)

Alfredo Sotomayor, Senior Audit Chemist

I like sushi. I have it regularly here in Madison, and when I travel to the ‘right’ places, I try to hit at least one sushi bar, sometimes, if I am lucky, in the company of others who share my passion. Yes, I know, many of you are repulsed by the idea of ingesting unprocessed tissue. But, like Ninotchka ², I also love raw vegetables. And where am I heading? Well, this not being “Bon Appetit”, or I Martha Stewart, you know that I will have to jump into the realm of data.

Sometimes I do like my food the way I like my data, the more unrefined, unprocessed, and raw, the better. Which does not mean I do not have preferences for how to process my data, just as I have distinct likes (and dislikes) about how my

¹ With apologies to Neneh Cherry.

² With apologies to the late Ernst Lubitsch and Greta Garbo.

food is prepared. But in this column I will deal with data in the raw.

UNCLEAR DEFINITIONS Our own Laboratory Certification and Registration Code (Chapter NR 149) defines raw data in about the same way as the NELAC Standards (we both used the same source, a document published by EPA titled “Good Automated Laboratory Practices”, GALP): *“Raw data means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of an analysis and are necessary for the reconstruction and evaluation of the analysis, which may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, and recorded information from automated collection systems.”*

There are two problems with this definition. The source, which attempted to establish guidelines for acceptable electronic encoding of data, primarily meant to give alternatives to the classic “hard copy” and to alert us about the obvious truth that copies are subject to manipulation. The definition clouds the issue by admitting that exact copies are raw, as if the plastic dessert replicas that some restaurants parade to entice us into ordering the real thing were edible.

The other problem, only implicit, with the definition is that an “original observation” is a well understood term. In the catalogue that follows of examples of types of media that can constitute “raw” data, the true nature of an “original observation” is lost, and thus the recording medium is confused with the content, the record, or should I say the CD, with the voice, the orchestra, or the band.

And yet we all understand the difference intuitively just as we can distinguish a bank statement from holding the corresponding balance in cash, a photograph and its subject, a folder and its content. One ‘is’, while the other is a record or a container of that ‘original’ experience.

RAWS BY ANY OTHER NAME A master tape or, ‘His Master’s Voice’? Perhaps we should use a different term to distinguish between an original observation and the medium (whatever the latter might be) used to record the observation. Calling a **chromatogram** with area counts ‘raw data’, contributes to the confusion. Analytical documents could be named unambiguously regardless of their content; for example, master or primary documents, for those directly out of an instrument; and secondary documents, for records of information at least once removed from the original observation or from the instrument.

A primary document would also contain the **first recorded instance** of a direct original observation. A secondary document would either reference the information contained in the primary document or would transform the same information into a different format or quantity, but in any event, would not be the first recorded instance of a direct original observation. **Copies** of either type of document, primary or secondary, would also not be the first recorded instance of an original observation, although they may still contain “raw” data. A chromatogram generated with a common data system after a calibration event would then be a primary document containing raw (and transformed) data.

In the next installment I will explore what I consider fundamental, primary, original observations or the other side of raw.

NEWS FLASHES

► On October 25, 1999 the long-awaited list of **NIST-approved Proficiency Testing providers** was published.

► Permittees will soon be notified by EPA of the **cancellation of the chemistry portion of DMRQA 19**. WDNR will inform permittees of the logistics of Study 20, once finalized.

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